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ARTICLE

Titanium Dioxide Coated Graphene Prepared by Pressure Sintering as Reinforcement in Aluminum Matrix Composites

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Abstract: Graphene nanoplatelets (GNPs) reinforced 7075Al nanocomposites were synthesized by pressure sintering method. A novel method for optimizing interfacial bonding through TiO₂ coated GNPs was proposed. The effects of TiO₂ coated GNPs on mechanical properties and microstructure of the aluminum matrix nanocomposites were investigated. Results show that the mechanical properties of the nanocomposites are improved by addition of TiO₂ coated GNPs, compared with those of nanocomposites with pure GNPs. The yield strength, ultimate tensile strength, and microhardness of the nanocomposites reinforced by TiO₂ coated GNPs are increased by 38.9%, 34.4%, and 20.1%, respectively, compared with those of the matrix. The improvement of the mechanical properties is attributed to the coating layer, which optimizes the interface bonding between the reinforcement and the matrix, thereby improving the efficiency of load transfer.

Key words: graphene nanoplatelets; TiO₂ coating; 7075Al alloy; composite

Graphene, a two-dimensional material consisting of six rings of sp²-hybridized carbon atoms, has been used as an important nano-size reinforcement in the field of structural and functional composites since 2004^[1-6]. It has attracted much attention due to the extremely high mechanical strength and excellent thermal and electrical properties. Compared with carbon nanotubes (CNTs), graphene has a larger surface area and lower production costs. The sheet structure of graphene makes it easier to disperse in the matrix, resulting in an excellent alternative reinforcement for composites. Currently, graphene has been extensively studied to enhance the mechanical and other properties of polymer or metal based composites^[7,8].

Aluminum and aluminum-based composites are widely used in automotive and aerospace fields due to their light mass and high corrosion resistance. With the development of industry, composites based on aluminum alloy are expected to demonstrate higher mechanical properties. Therefore, more and more scientists are trying to reinforce aluminum alloys with a strong, rigid, and lightweight phase^[9]. Recently, various

efforts have been made on graphene as a reinforcement of aluminum-based composites. Li et al^[10] fabricated a graphene nanosheets (GNSs)/Al nanocomposites by ball milling and vacuum hot pressing. The results show that the ultimate tensile strength (UTS) and yield strength (YS) are improved by 56.19% and 38.27%, respectively, due to the addition of GNSs, compared with those of pure Al. The microhardness of the nanocomposites is improved significantly as the content of GNSs increases. Zhang et al^[11] reported the similar results of the properties of Al5083 alloy after adding 1wt% graphene nanoplatelets (GNPs). It can be concluded that the high strengthening efficiency of the multi-layer GNPs may be attributed to shear lag (load transfer) effect and grain refinement effect. The nanosized Al₄C₃ formed in above composites could also act as a reinforcement. Moreover, the composite with 0.3wt% GNSs addition prepared by Wang et al^[12] exhibited an enhanced tensile strength, increasing by 62% compared with that of unreinforced matrix. However, some important problems of the graphene addition need to be solved. The main problem is the insufficient interface bonding

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between the nano-carbon material and the aluminum alloy, resulting in the fact that stress cannot be completely transferred from the matrix to the reinforcement during loading^[13].

In order to optimize the wettability and interfacial bonding between nano-carbon materials and alloy matrix and further improve the mechanical properties of the composite, surface modification techniques have been adopted for preparation of nano-carbon reinforcement. CNTs coated SiC nano-layers were prepared by Park et al^[14] using solid heating reaction method between silicon powders and CNTs ($C+Si \rightarrow SiC$). The results reveal that CNTs with SiC coating layer improve the wettability and bonding strength in Mg-based composite. Carbon nanofibers (CNFs) with copper coating were also used to enhance the mechanical properties of magnesium alloys^[15]. The results indicate that the improvement of wettability between Al and CNFs and non- Al_4C_3 is related to the Cu coating layer. There are a lot of reports about carbon nanotubes coated by metal or nano-ceramic particles as a reinforcement to strengthen the properties of metal matrix composites, suggesting the beneficial effect of coating on interfacial bonding and mechanical properties. However, the investigations on Al composites reinforced by coated graphene are rarely reported.

In this research, the 7075 aluminum nanocomposites reinforced by pure GNPs and TiO_2 coated graphene nanoplatelets ($TiO_2@GNPs$) were prepared by pressure sintering. The effects of the $TiO_2@GNPs$ and pure GNPs on the microstructure and the mechanical properties of the nanocomposites were characterized and investigated. Furthermore, the enhancement mechanisms of mechanical properties of nanocomposites were discussed.

1 Experiment

Commercial 7075 aluminum alloy powders (purity of 99.7%, particle size of 70~120 μm , Shenyang Nonferrous Metal Research Institute, China) were used as the matrix material in this research. Table 1 shows the chemical composition of 7075 aluminum powder. The graphene nanoplatelets (Nanchang Taiyang Nanocrystal Co., Ltd) of purity of 98.6wt%, average thickness of 6~8 nm, and particle size of 12~15 μm were used.

Firstly 0.2 g of acid-treated GNPs were placed in 20 mL ethanol and sonicated for 10 min (90 W). Then 0.5 mL of tetrabutyl titanate (TBT) and 10 mL of glycerol were added into the solution and sonicated for 5 min. The mixed solution was subsequently transferred to an autoclave and kept in muffle furnace at 160~200 $^{\circ}C$ for 12 h. Subsequently, the obtained solution was centrifuged to obtain the powder precipitates. The precipitates were washed with pure ethanol (99.7% purity) and then dried at 70 $^{\circ}C$ for 24 h. The obtained powders were then calcined in argon at 460~480 $^{\circ}C$ for 3 h.

Table 1 Chemical composition of 7075Al powder (wt%)

Zn	Mg	Cu	Fe	Si	Al
5.1~6.1	2.1~2.9	1.2~2.0	0.5	0.4	Bal.

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were used to investigate the microstructure of $TiO_2@GNPs$.

Fig. 1 illustrates the synthesis procedure of $TiO_2@GNPs/7075Al$ nanocomposites. As shown in the figure, 0.5wt% of $TiO_2@GNPs$ and 99.5wt% of 7075Al powder were put into corundum mixing tanks with corundum grinding balls with 5~20 mm in diameter. The initial mass ratio of ball to powder was 10:1. The mixing tanks were stirred using a planetary ball mill at 380 r/min for 24 h in argon atmosphere. And 1wt% stearic acid was added as a process control agent to prevent the excessive sticking and aggregation of the powders (cold welding) during ball milling. Then, the as-prepared nanocomposite powders were placed in a high-temperature graphite die and subjected to hot-press sintering at 595 $^{\circ}C$ for 2 h in the vacuum of 0.133 Pa under a uniaxial pressure of 28 MPa. The pressure sintering process parameters are also shown in Fig. 1. For comparison, the alloy without GNPs was also fabricated through the same process.

Nikon Eclipse MA200 optical microscope (OM) and NOVA NanoSEM 450 SEM were used to characterize the microstructures and the fracture surfaces of the nanocomposites. The nanostructures and interfaces were investigated by TEM (JEM-2100). Dimension of specimens for TEM observation prepared by traditional jet-polishing process was $\phi=3$ mm and thickness of 30 μm . Tensile tests were performed on a universal testing machine at room temperature with displacement rate of 0.2 mm/min to investigate the strengthening effect. Microscopic Vickers hardness tester (HVS 1000A) was used to obtain microhardness, according to EN-ISO 14577: 2003. The microhardness were measured at least five times for each specimen to obtain the average microhardness value.

2 Results and Discussion

2.1 Microstructure

Microstructure of GNPs is shown in Fig. 2a. Correspondingly, Fig. 2b and 2c exhibit the typical SEM and TEM images of the coated GNPs. It is evident that the surface of

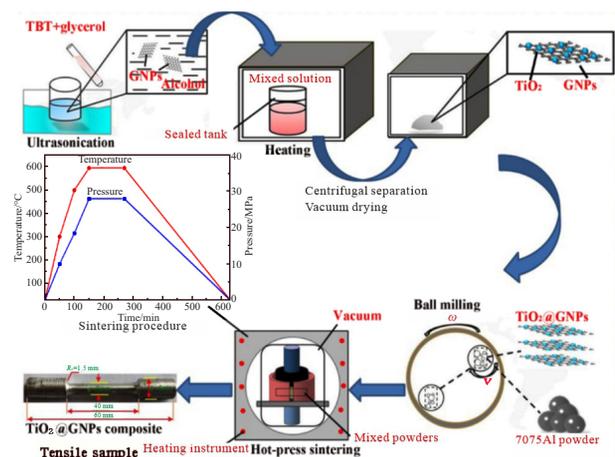


Fig.1 Schematic diagram of preparation of $TiO_2@GNPs/7075Al$ nanocomposites

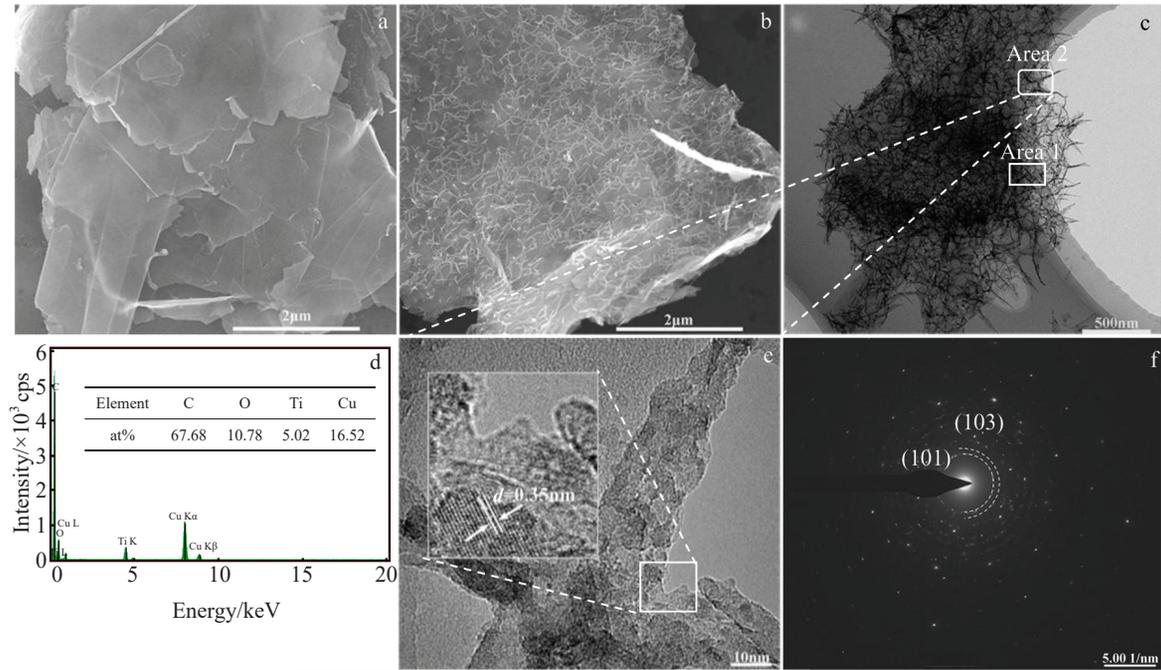


Fig.2 SEM images of GNPs (a) and TiO₂@GNPs (b); TEM image of TiO₂@GNPs (c); EDS spectrum of area 1 in Fig.2c (d); HRTEM image of area 2 in Fig.2c (e); SAED pattern of TiO₂ layer (f)

most GNPs is completely covered by a layer of white needle-like particles which appear to disorderly protrude outwards from the GNPs surfaces. The corresponding EDS spectrum indicates that the coating layer mainly consists of Ti and O. The relative concentration ratio between Ti and O is about 1:2. Meanwhile, the high resolution TEM (HRTEM) image and selected area electron diffraction (SAED) pattern show the d -spacings of the nanoparticles are 0.35 and 0.2894 nm, corresponding to the (101) TiO₂ plane in Fig. 2e^[16] and (103) TiO₂ plane in Fig. 2f, respectively. Therefore, it can be confirmed that titanium oxide is generated and coated on the surface of GNPs. In addition, it is observed that TiO₂ nanoparticles do not fall from the GNPs surface, even after sonication during the specimen preparation for TEM observation. This phenomenon indicates that a strong interfacial bond is generated between the nanoparticles and GNPs^[17].

Fig. 3 shows the irregularly shaped raw 7075Al particles with 70~120 nm in diameter. After milling, the morphologies of the 7075Al-0.9wt% GNPs powders are more spherical with a rounded flattening tendency than those of the raw 7075Al particles, as shown in Fig. 3b. The particle size is significantly smaller and the circled positions in Fig. 3b show the GNPs attached to Al particles. Meanwhile, it can also be seen from Fig. 3c that some individual GNPs with TiO₂ are embedded in the particles, indicating that GNPs with TiO₂ are uniformly dispersed in the surface of 7075Al particles or trapped in soft matrix by the ball milling process. In general, 7075Al powders are easily broken into small particles due to the shearing force by ball milling. The milling process causes these composite particles to collide with the inner surface of the containers and the balls and to generate enough energy to

change the shape of the powders. Eventually, the particles are crushed and flattened at the end of the milling^[18]. Then GNPs are incorporated into the 7075Al powders through plastic deformation during the milling process.

SEM images of the matrix alloy and GNPs/7075Al nanocomposites are shown in Fig. 4. It can be seen from Fig. 4b and 4c that the grain size of the alloy matrix becomes smaller after adding GNPs or TiO₂@GNPs. This phenomenon indicates that GNPs addition can refine the matrix grains because GNPs distributed at the grain boundaries can hinder the grain growth^[11]. In addition, some micropores can be observed on the surface of the alloy and GNPs/7075Al specimens due to the uneven grain refinement in the matrix. It can also be seen from Fig. 4b that agglomerations of GNPs appear at the grain boundaries, which is attributed to the poor dispersibility and wettability of GNPs in the matrix^[19]. As shown in Fig. 4c, no obvious reinforcement agglomeration occurs at the grain boundaries of the composites containing TiO₂@GNPs. This phenomenon mainly results from the optimization of the wettability of GNPs with the matrix, thereby promoting the combination of GNPs and alloy, which leads to a reduction in structural defects during the process.

Fig. 5 shows the XRD patterns of the prepared nanocomposites and the pure 7075Al alloy matrix. The diffraction peaks of Al(111), Al(200), Al(220), MgZn₂(112), MgZn₂(201), and AlCu₃(2012) can be clearly observed. Only a new weak diffraction peak of C(002) appears at $2\theta=27^\circ$, indicating the addition of GNPs in nanocomposites, because a trace number of GNPs and TiO₂ in the composites is difficult to detect by XRD with low electron density^[20].

Fig. 6 displays the interface structure of 7075Al nanocom-

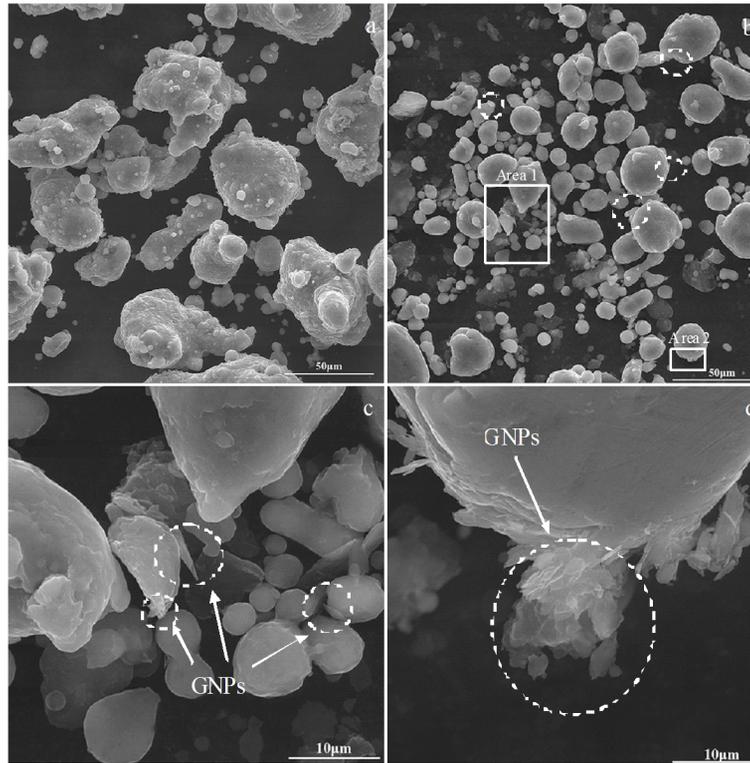


Fig.3 Morphologies of raw 7075Al (a) and 7075Al-0.9wt% GNPs (b) powders; magnified images of area 1 (c) and area 2 (d) in Fig.3b

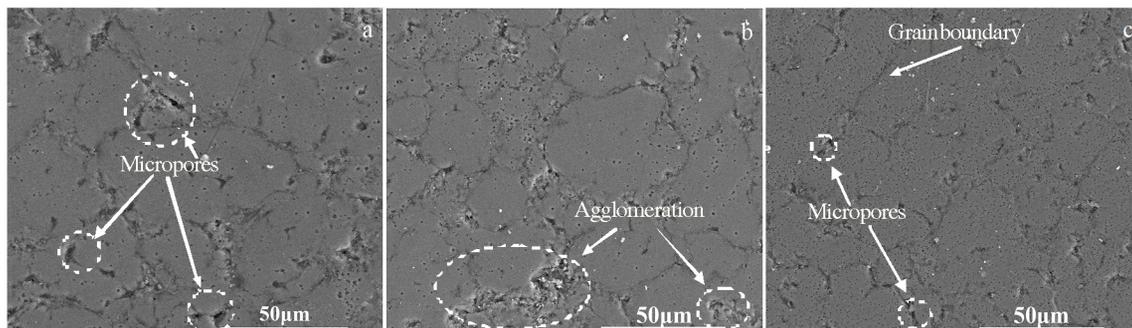


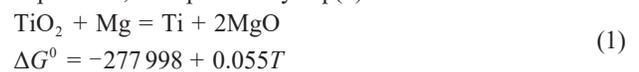
Fig.4 SEM images of 7075Al (a), GNPs/7075Al (b), and TiO_2 @GNPs/7075Al (c) nanocomposites

posite containing TiO_2 @GNPs. As shown in Fig. 6a, the carbon plane-structure is clearly visible and parallel to the matrix, indicating that GNPs maintain the multilayer structure after the preparation process at 595 °C. Moreover, GNPs and matrix are tightly attached to each other in the nanocomposite without obvious gaps or impurities, suggesting the strongly bonded interfaces between GNP and Al matrix^[21]. In addition, it can be observed from HRTEM images (Fig.6b and 6c) that there are some needle-like or sphere nanoparticles on the surface of GNPs. The d -spacing of these nanoparticles measured by SAED pattern (Fig. 6d) is 0.2319, 0.2133, and 0.1370 nm, corresponding to the $(2\bar{2}\bar{2})$, $(1\bar{3}2)$, and $(3\bar{2}\bar{2})$ TiO_2 planes, respectively. Therefore, it can be confirmed that the TiO_2 nanoparticles still exist and closely contact to the GNPs

surface.

It is worth noting that magnesium oxide (MgO) nanoparticles appear near the coating layer according to the results of Fig. 6d, i. e., a few MgO nanoparticles are formed and embedded at the interface. The d -spacing of MgO is 0.1196 and 0.0889 nm, corresponding to the (222) and (422) planes, respectively^[17].

It can be seen from Fig.7 that the generation of MgO can be attributed to the interfacial reaction between GNPs and TiO_2 nanoparticles, as expressed by Eq.(1)^[22]:



where ΔG^0 is free energy and T is the temperature. When the free energy is lower than 0, the reaction in Eq.(1) proceeds. In

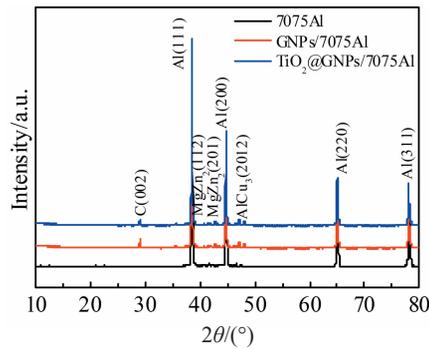


Fig.5 XRD patterns of 7075Al, GNP/7075Al, and TiO₂@GNPs/7075Al nanocomposites

this research, the highest sintering temperature provided by the equipment is 595 °C (868 K). The calculation results show that the free energy of reaction is less than zero, indicating the possibility of the reaction.

2.2 Mechanical properties

The mechanical properties of 7075Al and the nanocomposites reinforced by pure GNPs and TiO₂@GNPs are shown in Fig.8. YS and UTS of 7075Al matrix are only 198 and 253 MPa, respectively. However, the mechanical properties of GNP/7075Al and TiO₂@GNPs/7075Al nanocomposites are improved, and the increment in mechanical properties of TiO₂@GNPs/7075Al nanocomposite is more obvious than that of the other one. For GNP/7075Al nanocomposite, YS and

UTS are 252 and 310 MPa, respectively. For TiO₂@GNPs/7075Al nanocomposite, YS and UTS are 275 and 340 MPa, increased by 38.9% and 34.4% compared with those of the matrix, respectively. Furthermore, the microhardness of the GNP/7075Al and TiO₂@GNPs/7075Al nanocomposites remarkably increases to 1094 and 1162 MPa, respectively, which is obviously larger than that of matrix (967 MPa), increased by 20.1%. The GNPs addition can enhance the tensile properties and microhardness, and the further enhancement is attributed to the optimized interfacial bonding due to the presence of TiO₂ coating layer^[22,23].

Strengthening mechanisms of elongation refinement, dislocation strengthening, and stress transfer are commonly used to explain the improvement in mechanical properties, especially for the yield strength of GNPs/Al composites. In general, the huge difference in the coefficient of thermal expansion (CTE) between GNPs ($0.9 \times 10^{-6} \text{ K}^{-1}$) and Al alloy ($2.36 \times 10^{-5} \text{ K}^{-1}$) leads to the occurrence of prismatic punching at the interface, and then results in the strengthening of the nanocomposites. YS improvement of nanocomposites due to this mechanism^[21] can be calculated by Eq.(2) as follows:

$$\sigma_{\text{CET}} = 1.25Gb \sqrt{\frac{12\Delta T\Delta C f_v}{bd_p}} \quad (2)$$

where G is the shear modulus of Al ($2.6 \times 10^4 \text{ MPa}$); ΔT is the temperature difference; ΔC is the CET difference between GNPs and the matrix; f_v and d_p are the volume fraction and average diameter of GNPs, respectively; b is burger vector of matrix (0.286 nm).

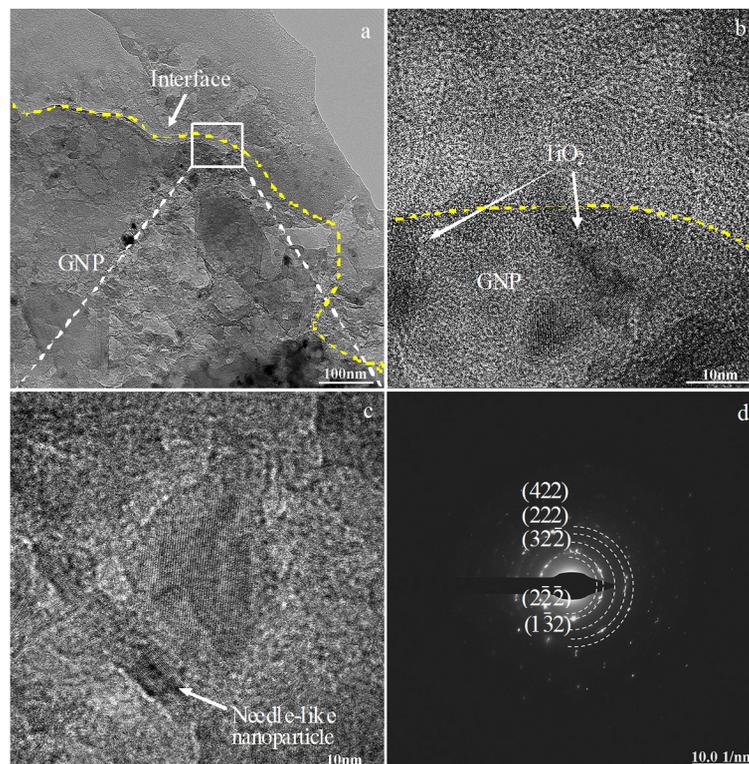


Fig.6 TEM (a) and HRTEM (b, c) images of TiO₂@GNPs/7075Al nanocomposites; SAED pattern of TiO₂@GNPs coating layer (d)

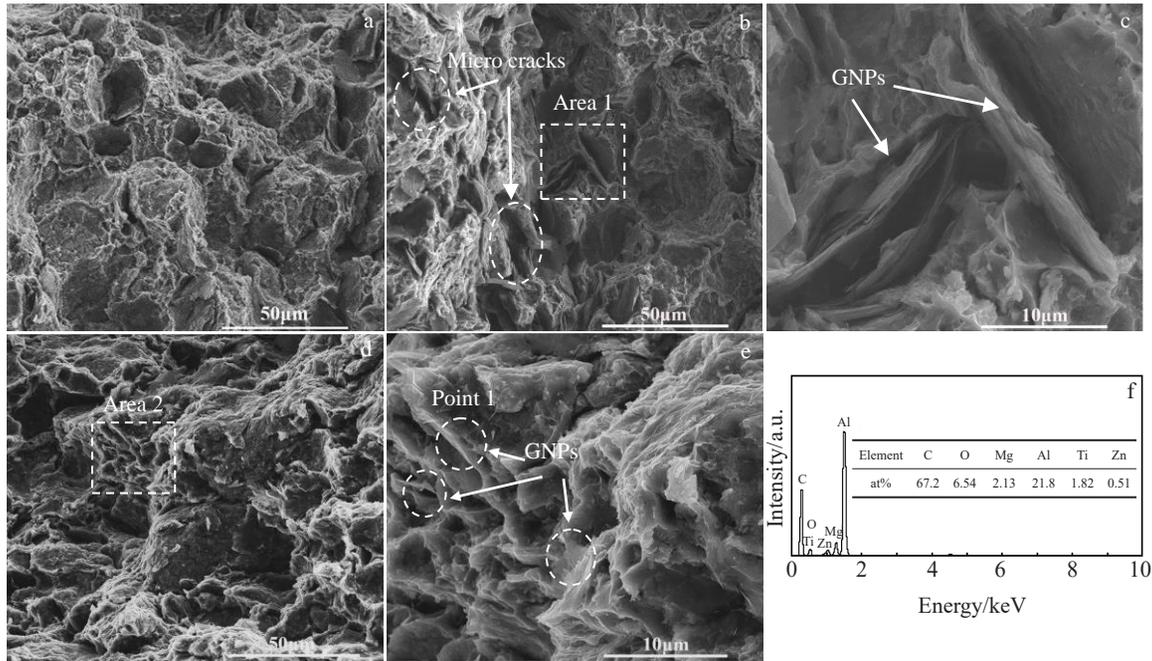


Fig.10 SEM fractographies of nanocomposites: (a) 7075Al, (b) GNPs/7075Al, (c) magnified image of area 1 in Fig. 10b, (d) TiO₂@GNPs/7075Al, and (e) detailed image of area 2 in Fig.10d; EDS spectrum of point 1 in Fig.10e (f)

properties of the GNPs reinforced Al-based nanocomposites. For GNPs/7075Al composites, GNPs aggregation occurs at the grain boundaries during the preparation process due to the poor wettability and interfacial adhesion of GNPs, as shown in Fig.4, which weakens the strengthening effect of GNPs on the mechanical properties of the nanocomposites. For TiO₂@GNPs/7075Al nanocomposites, the existence of TiO₂ coating layer improves the interface bonding between the reinforcement and Al matrix. Meanwhile, the products generated on the coating can result in an enhanced anchoring effect by the Al alloy matrix. These factors all facilitate efficient transfer of loads from the matrix to the GNPs^[23]. Hence, the mechanical properties of Al-based nanocomposites reinforced by TiO₂@GNPs are better than those of nanocomposites with uncoated GNPs.

The representative fracture surface characterizations of 7075Al and the prepared nanocomposites are shown in Fig.10. Correspondingly, the results of EDS analysis are shown in Fig.10f. The fracture surface of the matrix exhibits the mixed fracture and is characterized by the significant dimples and platforms (Fig. 10a). In nanocomposites reinforced by GNPs (Fig. 10b), some pullout of GNPs can be observed and the number of the platforms is decreased. However, two large cracks are visible on the fracture surface. These cracks show a propagating and growing trend under the bending load. When the reinforcement is TiO₂@GNPs, the fracture surfaces of specimen exhibit few cracks and the significant pullout of GNPs, indicating that the interfacial bonding between GNPs and 7075Al is improved due to the coating layer. EDS result reveals that these phases mainly consist of Al, C, O, Mg and Ti, confirming that the phases are GNPs and TiO₂.

It is worth noting that although the mechanical properties of prepared GNPs/Al nanocomposites are improved compared with those of the matrix, they are not particularly excellent. This is because the composites prepared by the current sintering process inevitably present a small number of micropores between grain boundaries. Therefore, how to improve the dispersion of GNPs in the matrix and the compactness of the composites should be investigated further. Hot extrusion and friction stir processing (FSP) can be used for process amelioration. The optimization of microstructure and performance, eliminating sintering defects, and preparing monolithic GNPs/Al composites with excellent mechanical properties should be focused on.

3 Conclusions

1) The addition of TiO₂ coated graphene nanoplatelets (TiO₂@GNPs) to 7075Al can effectively reduce the probability of reinforcement agglomeration at grain boundaries, compared with the nanocomposites reinforced by pure GNPs (GNPs/7075Al).

2) The mechanical performances and Vickers hardness of the nanocomposites are improved by TiO₂@GNPs addition, compared with those of GNPs/7075Al. The yield strength, ultimate tensile strength, and Vickers microhardness of the TiO₂@GNPs/7075Al nanocomposite increase by 38.9%, 34.4%, and 20.1%, respectively, compared with those of 7075Al.

3) The improvement in mechanical properties can be attributed to grain refinement, the huge difference in coefficient of thermal expansion, and load transfer mechanisms. The load transfer provides the maximum

contribution to composite strengthening. The existence of TiO₂ coating layers on the surface of GNPs strengthens the interfacial bonding; as a result the stress is effectively transferred from the matrix to GNPs during loading.

4) The addition of GNPs can significantly enhance the mechanical properties of 7075Al nanocomposites, and the TiO₂ particle layers coated on GNPs have a positive effect on the enhancement.

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压力烧结制备二氧化钛包覆的石墨烯增强铝基复合材料

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摘 要: 使用压力烧结方法制备了石墨烯纳米片 (GNP) 增强的 7075 铝基纳米复合材料, 提出了一种通过在 GNP 的表面涂覆二氧化钛 (TiO₂) 来优化界面结合的新工艺, 并对比了原石墨烯及具有包覆层石墨烯对铝基纳米复合材料的力学性能和微观结构的影响。结果表明, 与添加纯 GNP 相比, 添加具有 TiO₂ 涂层的 GNP 的纳米复合材料的力学性能提高。相比于基体, TiO₂ 包覆 GNP 增强的纳米复合材料的屈服强度、抗拉强度和显微硬度分别增加了 38.9%、34.4% 和 20.1%。性能的进一步改善是由于 TiO₂ 涂层优化了增强相与基体之间的界面结合, 从而提高了载荷传递的有效性。

关键词: 石墨烯纳米片; TiO₂ 涂层; 7075 铝合金; 复合材料

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